Los Alamos NATIONAL LABORATORY EST. 1943

ESHID-600920

Environmental Protection Division
Environmental Compliance Programs (ENV-CP)
PO Box 1663, K490
Los Alamos, New Mexico 87545
(505) 667-0666

National Nuclear Security Administration
Los Alamos Field Office, A316
3747 West Jemez Road
Los Alamos, New Mexico, 87545
(505) 667-5794/Fax (505) 667-5948

Date: SEP 17 2015
Symbol: ENV-DO-15-0248
LA-UR: 15-26357
Locates Action No.: N/A

Mr. John E. Kieling
Hazardous Waste Bureau
New Mexico Environment Department
2905 Rodeo Park Drive East, Building 1
Santa Fe, NM 87505

Dear Mr. Kieling:

Subject: Transmittal of Sampling and Analysis Plan for Unremediated Nitrate Salt Waste Containers at Los Alamos National Laboratory

The purpose of this letter is to transmit the enclosed sampling and analysis plan for the unremediated nitrate salt waste containers located at the Los Alamos National Laboratory within permitted storage at Technical Area (TA) 54, Area G. This document is being submitted to the New Mexico Environment Department- Hazardous Waste Bureau (NMED-HWB) as agreed upon by technical staff and staff from the US Department of Energy (DOE) and Los Alamos National Security, LLC (LANS), collectively the Permittees. This plan has been discussed during technical meetings with NMED-HWB, and is responsive to the ordered actions under the Administrative Compliance Order (HWB-14-20), issued by the NMED on December 6, 2014.

The sampling and analysis plan has been drafted to include onsite sampling and analysis of unremediated nitrate salts to provide additional chemical composition data on the nitrate salt waste stream. The plan was drafted utilizing available US Environmental Protection Agency (EPA) guidance and onsite analytical reports for other nitrate salt-bearing waste. Onsite analysis is necessary in this case because the Permittees have been unsuccessful at identifying a safe and effective method for shipping previous samples of like material. The Permittees will continue to assess off-site facilities, shipment methods, and other avenues to obtain independent data from an EPA-certified laboratory as well as proceed with onsite analysis as planned.

If you have comments or questions regarding this submittal, please contact Mark P. Haagenstad at (505) 665-2014 or Gene E. Turner at (505) 667-5794.

Sincerely,

Alison M. Dorries
Division Leader
Environmental Protection Division
Los Alamos National Security, LLC

Sincerely,

Gene E. Turner
Environmental Permitting Manager
National Security Missions
Los Alamos Field Office
U.S. Department of Energy

AMD:GET:MPH:LRVH/lm

Enclosures: (1) Sampling and Analysis Plan, Unremediated Nitrate Salt Waste Containers at Los Alamos National Laboratory

Cy: Ryan Flynn, NMED, Santa Fe, NM, (E-File)
Kathryn Roberts, NMED, Santa Fe, NM, (E-File)
Dave Cobrain, NMED/HWB, Santa Fe, NM, (E-File)
Neelam Dhawan, NMED/HWB, Santa Fe, NM, (E-File)
Siona Briley, NMED/HWB, Santa Fe, NM, (E-File)
Kimberly Davis Lebak, LASO-OOM, (E-File)
Christine M. Gelles, EM-LA, (E-File)
David Nickless, EM-WM, (E-File)
Lisa Cummings, LASO-OC, (E-File)
Gene E. Turner, LASO-NS-LP, (E-File)
Kirsten M. Laskey, LASO-SUP, (E-File)
Jordan Arnswald, LASO-NS-PI
Craig S. Leasure, PADOPS, (E-File)
Amy E. De Palma, PADOPS, (E-File)
Randall M. Erickson, ADEP, (E-File)
Cheryl D. Cabbil, ADNHHO, (E-File)
Michael T. Brandt, ADESH, (E-File)
Raeanna R. Sharp-Geiger, ADESH, (E-File)
Alison M. Dorries, ENV-DO, (E-File)
Enrique Torres, ADEP, (E-File)
Leslie K. Sonnenberg, EWMO-DO, (E-File)
Andrew R. Baumer, ADEP-PDO, (E-File)
Brian P. Colby, WDD, (E-File)
Paul N. Newberry, WD-SRS, (E-File)
David E. Frederici, WD-WPE, (E-File)
Kenneth M. Hargis, WD-WPE, (E-File)
Mark P. Haagenstad, ENV-CP, (E-File)

Cy (continued):
Deborah L. Guffee, SI-DC, (E-File)
Yvette S. Branch, SI-DC, (E-File)
Luciana Vigil-Holterman, ENV-CP, (E-File)
Deborah K. Woitte, LC-ESH, (E-File)
lasomailbox@nnsa.doe.gov, (E-File)
emla.docs@em.doe.gov, (E-File)
locatesteam@lanl.gov, (E-File)
env-correspondence@lanl.gov, (E-File)
epccat@lanl.gov, (E-File)
rcra-prr@lanl.gov, (E-File)

Environmental Protection Division
Environmental Compliance Programs (ENV-CP)
PO Box 1663, K490
Los Alamos, New Mexico 87545
(505) 667-0666

National Nuclear Security Administration
Los Alamos Field Office, A316
3747 West Jemez Road
Los Alamos, New Mexico, 87545
(505) 667-5794/Fax (505) 667-5948

Date: SEP 17 2015
Symbol: ENV-DO-15-0248
LA-UR: 15-26357
Locates Action No.: N/A

Mr. John E. Kieling
Hazardous Waste Bureau
New Mexico Environment Department
2905 Rodeo Park Drive East, Building 1
Santa Fe, NM 87505

Dear Mr. Kieling:

Subject: Transmittal of Sampling and Analysis Plan for Unremediated Nitrate Salt Waste Containers at Los Alamos National Laboratory

The purpose of this letter is to transmit the enclosed sampling and analysis plan for the unremediated nitrate salt waste containers located at the Los Alamos National Laboratory within permitted storage at Technical Area (TA) 54, Area G. This document is being submitted to the New Mexico Environment Department- Hazardous Waste Bureau (NMED-HWB) as agreed upon by technical staff and staff from the US Department of Energy (DOE) and Los Alamos National Security, LLC (LANS), collectively the Permittees. This plan has been discussed during technical meetings with NMED-HWB, and is responsive to the ordered actions under the Administrative Compliance Order (HWB-14-20), issued by the NMED on December 6, 2014.

The sampling and analysis plan has been drafted to include onsite sampling and analysis of unremediated nitrate salts to provide additional chemical composition data on the nitrate salt waste stream. The plan was drafted utilizing available US Environmental Protection Agency (EPA) guidance and onsite analytical reports for other nitrate salt-bearing waste. Onsite analysis is necessary in this case because the Permittees have been unsuccessful at identifying a safe and effective method for shipping previous samples of like material. The Permittees will continue to assess off-site facilities, shipment methods, and other avenues to obtain independent data from an EPA-certified laboratory as well as proceed with onsite analysis as planned.

ENCLOSURE 1

Sampling and Analysis Plan, Unremediated Nitrate Salt Waste Containers at Los Alamos National Laboratory

ENV-DO-15-0248

LA-UR-15-26357

Date: SEP 1 7 2015

Sampling and Analysis Plan

Unremediated Nitrate Salt Waste Containers at Los Alamos National Laboratory

Los Alamos National Laboratory

LA-UR-15-26357

August 2015

Blank page

Approvals

[signature] 9/9/15

Michael T. Brandt, Associate Director
Environmental, Safety, and Health
Los Alamos National Laboratory

Date

[signature] for R. Erickson 9/8/15

Randall M. Erickson, Associate Director
Environmental Programs
Los Alamos National Laboratory

Date

Antoinette Taylor 9/9/15

Nancy N. Sauer, Associate Director
Chemistry, Life & Earth Sciences
Los Alamos National Laboratory

Date

Blank page

1.0 SITE INFORMATION/BACKGROUND

This sampling plan has been developed in support of the resumption of transuranic (TRU) waste–processing activities at Los Alamos National Laboratory (LANL). There are 60 remediated and 29 unremediated nitrate salt–bearing waste containers currently located at LANL that must be treated before they are shipped to the Waste Isolation Pilot Plant (WIPP). Generation of these wastes originally occurred as a result of plutonium recovery processing at Technical Area 55. Remediation of the original waste stream through the addition of organic cat litter resulted in remediated nitrate salt–bearing waste containers, with one of the remediated containers (68660) responsible for the radiation release at WIPP. The unremediated nitrate salt waste containers represent the original waste stream generated from plutonium recovery processing.

Containers of remediated and unremediated nitrate salt waste are characterized as exhibiting the U.S. Environmental Protection Agency (EPA) Hazardous Waste Number D001 for ignitability (remediated and unremediated) and D002 for corrosivity (unremediated with liquids only). Because these hazardous waste numbers cannot be accepted for disposal at WIPP, waste treatment of both remediated and unremediated nitrate salt waste must be conducted before certification, shipment, and disposal. It has been determined that before a compliant treatment methodology is selected for all the nitrate salt–bearing waste containers located at LANL, it is necessary for the U.S. Department of Energy (DOE) and the Los Alamos National Security, LLC (LANS), collectively, the Permittees, to perform additional waste characterization. Waste characterization efforts under this plan are being conducted as part of the Permittees' recharacterization efforts as required by the LANL Hazardous Waste Facility Permit, Section 2.4.7(4). To obtain more acceptable knowledge information and gain further characterization documentation for this waste stream in accordance with the requirements of Title 40 of the Code of Federal Regulations (CFR) 264.13, sampling and analysis will be conducted. The opening of waste storage containers for the purpose of sampling the contained waste is consistent with the requirements of the LANL Hazardous Waste Facility Permit.

Unremediated nitrate salt waste located at LANL is in 55-gal. containers that are overpacked into 85-gal. containers. The contents of 55-gal. unremediated nitrate salt waste containers are bags of nitrate salt mixtures that may or may not have deteriorated. All the containers confirmed to have bags within the 55-gal. container include a 90-milliliter (mL) rigid liner as well as a lead liner in varying conditions, as can be determined by the real-time radiography (RTR) videos reviewed. The containers also contain liquids that are either confined in the bags, are partially absorbed throughout the container, or are free liquid within the liner of the container.

2.0 PROJECT DESCRIPTION AND GOALS

This plan is designed to outline the process by which the Permittees provide documentation to confirm that remediated nitrate salts are adequately characterized and that surrogate wastes, which contain a hazardous component of the waste but are not radioactive, can be developed to bound the most thermally sensitive nitrate salt-sorbent combinations in support of process parameter selection for processing the remediated nitrate salt waste stream. The sampling and analysis plan includes the following:

(1) A list of representative constituents (see Table 1) that shall be utilized to select the analytical methods.

(2) A plan for obtaining samples within each of the waste containers chosen for sampling as detailed in Section 2.4.

(3) The type of samples to be collected (e.g., solid and liquid) and the rationale for the selection of the sample type.

(4) Sampling methods including a description of the methods and procedures that will be used to collect each type of sample as specified in Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846) (EPA 1986) or as modified to meet internal safety procedures.

(5) A description of the analytical methods that will be used to measure constituent concentrations and other characteristics of the waste (see Table 2).

(6) This sampling and analysis plan includes a description of the QA/QC procedures that include, but are not limited to, a description of all sample handling, labeling, and chain-of-custody procedures.

Because the unremediated nitrate salt waste containers do not pose the same safety hazards as remediated nitrate salt–bearing waste, unremediated nitrate salt waste containers were selected for sampling and analysis to provide further characterization information on the nitrate salt-bearing waste streams (LANL 2015a). Sampling and analysis of the contents of these containers will provide additional waste characterization information by acceptable knowledge for the portion of the MIN02-V.001 waste stream for containers that include nitrate salt waste. The results will be used (1) to supplement the characterization of the nitrate salt waste stream in its entirety, (2) to ensure that surrogates used for treatment feasibility testing are as representative of the waste stream as possible, and (3) to provide additional data regarding the salt mixtures and liquids within the nitrate salt waste that will be used as evidence to further understand the potential for chemical reactivity when mixed with sorbents.

In summary, the objective of sampling and analyses conducted as a result of this plan is to obtain useful information regarding the constituents and mixtures of salts and liquids within the unremediated nitrate salt waste containers remaining in storage at LANL to support acquiring acceptable knowledge about the waste stream for the purposes of evaluating treatment and disposal pathways.

2.1 Data Limitations and Objectives

This sample collection campaign cannot be considered to be "representative sampling" as defined by 40 CFR 260.10 because of the limited number of containers available, the variability of the waste form in the containers, and the lack of consistency/completeness of historical documentation. Therefore, the resulting analyses will be used to supplement historical documentation as well as provide further information about the waste stream.

Samples selected will be analyzed to confirm information known about the unremediated nitrate salt waste stream and to obtain further information on potential variability of the salt mixtures and the composition of the liquid within the waste containers. The data obtained will be useful in characterizing the average mixtures of the composition of the nitrate salts and any difference between the solid salts and liquids within the containers. Note, the data obtained from this sampling campaign may not exhibit all bounding properties of the waste stream. In the event that the sampling analyses results indicate wide variability of the basic components of the waste (e.g. unexpected constituents, debris, or polarized data), the need for more sampling will be evaluated.

It is recognized that an independent off-site analytical laboratory with EPA certification is preferable, however, due to radiological concentrations the Permittees were unsuccessful at identifying a safe and effective method for shipping samples collected from unremediated nitrate salt waste containers in 2014. Therefore, in the interest of obtaining sample analytical data, analyses will be conducted at an on-site analytical laboratory at this time. The Permittees will continue to assess off-site facilities, shipment methods, and other avenues to obtain independent data. If a path for this is found the Permittees will utilize the archive samples discussed in Section 4. All samples collected from empty or full containers of this waste stream previously have been analyzed on-site by the Actinide Analytical Chemistry Group. The constituents that can be identified using analyses available on-site are listed in Table 1. Instrumentation to identify these constituents as well as instrumentation that will be used to identify other characteristics of the waste for additional waste characterization data is outlined in Table 2.

Treatment methodology test planning is currently underway and as part of testing, surrogate waste samples will be created and sent to an off-site analytical laboratory for testing in accordance with Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846) (EPA 1986). The average mixture concentration and characterization of the liquids in the unremediated nitrate salt waste containers will be used in this effort as information to develop a surrogate for testing. Additional information on treatment methodology testing will be provided to the New Mexico Environment Department (NMED) for review as it is drafted.

2.2 Information Required for Sample Selection Assessment

The information available for the sample selection assessment included historical waste generator documentation and RTR video or RTR video documentation.

2.3 Sample Selection Boundaries

Of the original 272 nitrate salt parent waste containers, 29 waste containers are available on-site at LANL for potential sampling. The following list outlines other boundary conditions that exist for sample collection activities.

a. All the waste is containerized and presents physical access challenges for sampling.
b. All the waste containers present radiological exposure concerns.
c. Sample collection equipment (and all other handling equipment) must be nonsparking because the waste stream has been characterized as ignitable waste.
d. Historical documentation and RTR video are not always consistent when it comes to the quantity of the contents within a given waste container.
e. Two of the 29 waste containers include cans of material determined to be uncemented nitrate salt waste. Because the waste stream has been characterized as ignitable waste, additional safety concerns are present for opening individual cans within a waste container.
f. For two other waste containers, RTR video is not available and limited information is included on the RTR log sheet documentation available for the waste containers; therefore, there are more unknown variables if these containers are sampled.
g. Liquid within the containers has the potential to be acidic, and the nature of the salts themselves has the potential to corrode steel (EPA Hazardous Waste Number D002), so additional precautions are required when sampling.

2.4 Sample Selection Process

Judgmental sampling (EPA 2002a and EPA 2002b) was chosen as the most logical strategy for selecting containers to sample. Judgmental sampling can be used to generate rough estimates of the average concentration of a property that is typical to a waste stream and can help to assess the usefulness of samples drawn from a small portion of a waste stream. Limitations of the judgmental sampling strategy are based on knowledge of the waste. The approach is not probability-based, so inference to the general population can be difficult if results are not as expected, and it cannot reliably determine estimates of variability. In this case, judgmental sampling is acceptable because information for the waste from the plutonium recovery process is known, and the purpose of the sampling campaign is limited to gaining additional information for the various salt constituents as well as characterization data for the liquid associated with those salts.

In selecting containers to be sampled, each of the following questions was assessed for each of the 29 containers.

a. From the RTR video review, does it appear that there are available locations within the container for sample collection of the nitrate salt solids?
b. Is liquid present within the container? Is it accessible for sampling?
c. Does the waste-generating process identified in the available waste documentation represent a waste-generating process not already covered in the containers already selected?
d. Is the waste generation date (year) for the container representative of a time frame not already covered in the containers already selected?
e. Are the radiological characteristics of the selected waste containers representative of the range observed in the total (29) containers available for sampling?

Based on analysis of the available containers and with consideration of responses to the questions above, nine waste containers were selected for sampling, with judgmental reasoning as to why they were chosen documented in Table 3. One alternate has been identified in the event that during sample collection planning another container from that waste generation year must be eliminated because of a safety hazard identified during sample collection planning activities. Additionally, the generation years 1984 and 1985 do not have any containers that could be chosen to be sampled safely at this time. Therefore, the containers for which there is no RTR video will be sent to RTR and evaluated again for potential sampling. In Table 3 these two containers are marked “Potential”.

Composite sampling is a physical averaging process that is recommended for sample collection from unremediated nitrate salt waste containers because the technique tends to produce samples containing constituents that are more normally distributed than grab samples (EPA 2002a and EPA 2002b). Additionally, composite sampling can be used to provide better representation of the salt mixtures within the waste containers without the addition of individual analysis and with the same cost, time, and risk to worker.

To determine whether any stratification of the contents within a waste container is occurring, a profile sample collection system is proposed for each of the waste containers. Four samples are proposed to be collected from full waste containers: three solid composite samples and one liquid sample. None of the waste containers available to select for sampling are full; therefore, not all samples will be obtainable. The general sampling collection strategy for a full container is as follows.

1. Three grab samples of solids from approximately the top 10 inches (in.) of the waste container will be composited into one waste sample. A grab sample from two sides (near the inner container liner) and one grab sample from the middle of the waste container should be collected and composited into one sample container. These composite samples will not be field mixed. For safety, mixing will be conducted at the analytical laboratory.
2. Three grab samples of solids from approximately the mid-level (11–21 in.) of the waste container will be composited into one waste sample. A grab sample from two sides (near the inner container liner) and one grab sample from the middle of the waste container should be collected and composited into one sample container. These composite samples will not be field mixed. For safety, mixing will be conducted at the analytical laboratory.
3. Three grab samples of solids from approximately the lower-level (22 in. to bottom) of the waste container will be composited into one waste sample. A grab sample from two sides (near the inner container liner) and one grab sample from the middle of the waste container should be collected and composited into one sample container. These composite samples will not be field mixed. For safety, mixing will be conducted at the analytical laboratory.
4. A liquid sample will be collected if sufficient liquid is present. Available RTR documentation will be used to focus liquid sampling within the container.

Waste containers chosen for sampling, the proposed location (stratification) for waste sampling, waste sampling type(s) and justification for container selection is included in Table 3. As Table 3 outlines, the unremediated nitrate salt waste containers are no more the 2/3 to 3/4 full. Therefore, the sampling strategy changed so that three solid composite samples as detailed above will be attempted for waste containers that are at least 2/3 full and 2 solid composite samples will be attempted for waste containers approximately 1/2 full.

3.0 SAMPLE COLLECTION

This sampling and analysis plan identifies the specific sampling and analysis requirements for the remaining 29 unremediated nitrate salt waste containers at LANL and describes the sampling, analysis, and quality assurance/quality control (QA/QC) methods that will be used to demonstrate the Permittees have provided the information outlined in Section 2 of this plan. Table 3 of this plan outlines the desired samples within each of the containers selected for sampling, proposed positions of samples to be collected from the container, and the container numbers selected for sampling. The locations include a proposed 22 solid composite samples and 9 liquid samples from 9 waste containers, depending on as-found conditions and capability to safely obtain samples.

3.1 Sampling Activities

Before waste containers are opened to collect samples of the waste, headspace-gas sampling will be conducted to ensure gases within the containers are within a normal range and are safe to be opened.

Sampling activities will be conducted to obtain liquid samples and solid composite samples from unremediated nitrate salt waste containers to meet the objectives described in Section 2. All samples will be collected and analyzed in accordance with the procedures in Section 4 of this plan.

3.2 Sample Collection Procedures

Samples will be collected in accordance with the procedures identified in this SAP which incorporates guidance from the EPA (EPA 1986, EPA 2002a, and EPA 2002b); DOE (DOE 1995); and other LANL-implemented and -approved procedures for sampling and analysis as implemented by waste management personnel.

Before samples are collected, the sampling plan must be approved by the Associate Director of Environmental Programs; the Associate Director for Environmental, Safety and Health; and the Actinide Analytical Chemistry Group. All tools used for sample collection will be nonsparking tools because the waste being sampled is characterized as ignitable waste (EPA Hazardous Waste Number D001). Sample tools will be reused for samples within a single composite or for replicate samples only. Separate tools will be used for individual samples; therefore, a description of decontamination of sampling equipment is not included herein.

3.2.1 Solid Sampling

If possible, solid samples will be collected from the top level, middle level, and bottom level of each of the 55-gal. waste containers chosen for sampling and outlined in Table 3. Samples of the waste within the container will be collected using a scoop or powder/granule thief made of polypropylene, PEEK (polyether ether ketone), polyethylene, or Teflon.

Samples will be composited in a 120 mL or larger Teflon container, sealed tightly with at least 30 to 40 grams (g) of material.

3.2.2 Liquid Sampling

If possible, liquid samples will be collected from all of the 55-gal. waste containers selected for sampling as outlined in Table 3. Liquid samples will be collected using long-tipped syringes or pipettes made with polypropylene, polyethylene, or Teflon; or a peristaltic pump.

Samples will be collected in a 120mL or larger Teflon container, sealed tightly with at least 5 mL to 50 mL of liquid.

3.3 Sample Documentation

Sampling personnel will complete and maintain records to document sampling and analysis activities. Sample documentation will include sample identification numbers, chain-of-custody forms, analysis requested, sample logbooks detailing sample collection activities, and shipping forms (if necessary).

3.3.1 Chain of Custody

Chain-of-custody forms will be maintained by sampling personnel until the samples are relinquished to the analytical laboratory. This will ensure the integrity of the samples and provide for an accurate and defensible written record of the sampling possession and handling from the time of collection until shipment to the laboratory for analysis. One chain-of-custody form may be used to document all samples collected from a single sampling event or day. The sample collector will be responsible for the integrity of the samples collected until they are properly transferred to another person. The EPA considers a sample to be in a person's custody if it is

a. in a person's physical possession;
b. in view of the person in possession; or
c. secured by that person in a restricted access area to prevent tampering.

The sample collector will document all pertinent sample collection data. Individuals relinquishing or receiving custody of the samples will sign, date, and note the time on the analysis request and chain-of-custody form. A chain-of-custody form must accompany all samples from collection through laboratory analysis. The analytical laboratory will return the completed chain-of-custody form to the facility, and it will become part of the permanent sampling record documenting the sampling efforts. The LANL information management system for the Actinide Analytical Chemistry Group is Labware. Samples are logged and distributed into Labware for analysis. The Labware database tracks the sample while it was being analyzed and will serve as the chain of custody once the analytical laboratory receives it.

3.3.1.1 Sample Labels and Custody Seals

A sample label will be affixed to each sample container. The sample label will include a unique sample identification number that will correspond to the information found in the sample logbook. A custody seal will be placed on the final outer bag that contains the sample container to detect unauthorized tampering with the samples. The labels with the unique identification number will be affixed by the sample collector before he or she enters the radiological area where the samples will be collected.

3.3.2 Sample Logbook

All pertinent information on the sampling effort must be recorded in a bound logbook. The information must be recorded in ink and any cross-outs must be made with a single line with the change initialed and dated by the author. The sample logbook will include the following information:

a. container number
b. the sample location (including position within container)
c. suspected composition
d. sample identification number
e. volume/mass of sample taken
f. purpose of sampling
g. description of sample point and sampling methodology
h. date and time of collection
i. name of the sample collector
j. sample destination and how it will be transported
k. observations and
l. name(s) of personnel responsible for the observations

3.4 Sample Handling and Storage

Samples will be collected and containerized in appropriate precleaned sample containers. Samples will be triple-contained (e.g., vial or container, Ziploc bag, then another Ziploc bag). The final outer bag will be decontaminated to free-release radiological levels, and a custody seal will be placed on the outer bag. Sample containers and quantity of sample required for all analyses are described in Section 3.2.1 above.

3.5 Packaging and Transportation of Samples

All packaging and transportation activities will meet safety expectations, QA requirements, DOE orders, and relevant local, state, and federal laws (including 10 CFR and 49 CFR). Appropriate facility documents establish the requirements for packaging design, testing, acquisition, acceptance, use, maintenance, and decommissioning and for on-site and intra-site shipment preparation and transportation of general commodities, hazardous materials, substances, waste, and defense program materials. This sampling and analysis plan does not include the transportation of waste samples off-site.

4.0 SAMPLE ANALYSIS REQUIREMENTS

Samples will be analyzed for the constituents listed in Table 1. These constituents are determined to be applicable constituents that will aid in determining the variability of the salt contents within the unremediated nitrate salt wastes. If new information is discovered, the list shall be amended to include additional constituents for sampling and analysis. Samples will be analyzed by the Actinide Analytical Chemistry Group using the methods and instrumentation outlined in Table 2.

4.1 Analytical Laboratory Requirements

The analytical laboratory at the Chemical Metallurgy Research (CMR) Facility will perform the detailed qualitative and quantitative chemical analyses specified in Section 4.2. The analytical laboratory will have

a. a documented comprehensive QA/QC program;
b. technical analytical expertise;
c. a document control/records management plan; and
d. the capability to perform data reduction, validation, and reporting.

The selection of the analytical testing methods identified in Table 2 is based on the following considerations:

a. the physical form of the waste
b. constituents of interest and
c. information requirements (e.g., waste classification)

4.2 Quality Assurance/Quality Control

All sampling and analysis will be conducted in accordance with the QA/QC procedures and methods defined by Actinide Analytical Chemistry Group procedures because the samples will be analyzed by an internal laboratory. These QA/QC procedures follow requirements outlined in QA-1 (Actinide Analytical Chemistry Quality Assurance Program, C-AAC 2015). Laboratory QC is required to ensure continuing precision, accuracy, and sensitivity of analytical measurements are consistent with customer requirements. Acceptance limits for QC measures must be specified as part of the customer’s overall data quality objectives, and corrective actions must be taken when these limits are not met. Examples of such quality measures should include, but are not limited to,

a. instrument calibration; and
b. internal QC samples, such as surrogate samples, spiked samples, replicates, duplicates, blanks, reference control samples, and standards.

The Actinide Analytical Chemistry Group management and workforce are committed to good professional practices and to the quality of its testing, services, and analytical chemistry results to customers. The Actinide Analytical Chemistry Group shall be familiar with and implement this QA program and all associated quality procedures that make up the program to ensure customer quality requirements are met. Information on the evaluation of the QC results is also described below.

4.2.1 Analytical Laboratory Quality Control Samples

QA/QC considerations are an integral part of analytical laboratory operations. Laboratory QA ensures that analytical methods generate data that are technically sound and statistically valid and that can be documented. QC procedures are the tools employed to measure the degree to which these QA objectives are met. Archive samples will be split out from field collected samples and will be kept by the analytical laboratory and analyzed if questions about the original analyses arise or will be sent off-site if a location can be found to take the sample and shipment of the sample can be arranged.

4.3 Data Reduction, Verification, Validation, and Reporting

Analytical data generated by the activities described in this sampling plan will be verified and validated. Data reduction is the conversion of raw data to reportable units, transfer of data between recording media, and computation of summary statistics, standard errors, confidence intervals, and statistical tests.

4.4 Data Reporting Requirements

Analytical results will include all pertinent information about the condition and appearance of the sample as received. Analytical reports will include

a. a summary of analytical results for each sample;
b. results from QC samples such as blanks, spikes, and calibrations;
c. reference to standard methods or a detailed description of analytical procedures; and
d. raw data printouts for comparison with summaries.

The laboratory will describe the analysis in sufficient detail so that the data user can understand how the sample was analyzed.

5.0 ANALYTICAL DATA USE AND STATISTICAL ANALYSIS

The data obtained from the samples will be used to characterize the composition of the material in the containers by calculating estimated means and associated standard errors for each of the species that is analyzed. Estimated means will be computed separately for the composite and the liquid samples. In the event that sample analyses results indicate wide variability of the basic components of the waste, the need for more sampling will be evaluated.

5.1 Calculation of Mean and Standard Error of Mean for Liquid Samples

Means for the liquid samples will be computed by averaging over the values obtained for each container. The associated standard deviation will also be computed using the values obtained for each container. The standard deviation can then be used to compute the standard error of the mean by dividing the standard deviation of the individual sample values by the square root of n_L, where n_L is the number of liquid samples obtained.

Estimated mean: $Xbar = \sum x_i / n_L$, $i=1,\ldots, n_L$.

Standard error of mean: $S=s/\{\text{square root}(n_L)\}$, where s is the standard deviation of the values,

$s=\{\sum (xi-Xbar)**2\}/(n_L -1)$.

5.2 Calculation of Mean and Standard Error of Mean for Composite Samples

Means of the composite samples will be calculated by first computing an average value over the composites within a container for each container and then averaging over the individual container means to obtain an overall estimated mean.

> Let y_{ij}=the measured value from container i, composite j. Then Yi, the mean of the composites in the ith container, is given by $\sum y_{ij}/m_i$, where m_i is the number of composite samples from the ith container.
>
> The overall mean is given by Ybar = $\sum Yi/ n_C$, where n_C is the number of containers from which one or more composite samples were obtained.

The standard error of the estimated overall mean for the composite samples can be obtained using a pooled variance estimate incorporating information about variability from each container for which there are at least two composite samples, assuming a common variance across containers.

Let s_i be the estimated standard deviation of the composite values for the ith container, i=1,…, k, where k is the number of containers with at least two composite samples. (A standard deviation calculation requires at least two data values, so variability information will be obtained only from containers with at least two composite samples.)

> Then the pooled variance estimate is given by
>
> $s_p^2=\sum (m_i-1)s_i^2/\{\sum (m_i -1)\}= \{(m_1-1)s_1^2+(m_2-1)s_2^2 +\ldots+ (m_k-1)s_k^2\}/(m_1+ m_{2+ \ldots+} m_{k)} – k)$.
>
> The standard error S of the overall mean, Ybar, is given by
>
> $S=ms_p^2/ n_C$, where $m =\sum (m_i)$ is the total number of composite samples from all the containers.

6.0 RECORDS

Records generated as a result of this sampling and analysis campaign will be kept as part of the Facility Operating Record in accordance with LANL Hazardous Waste Facility Permit (NMED 2010).

7.0 WASTE MANAGEMENT

By removing waste for analysis from unremediated nitrate salt waste containers, the owner/operator may become a generator of hazardous waste. The generator shall control, handle, characterize, and dispose of all wastes generated during sampling activities in accordance with facility waste management procedures and in compliance with applicable state, federal, and local requirements per P409, *LANL Waste Management* (LANL 2015b), and the LANL Hazardous Waste Facility Permit (NMED 2010).

Waste generated may include, but is not limited to, personnel protective equipment and disposable sampling equipment that may be contaminated with waste that exhibits the EPA Hazardous Waste Numbers for ignitability (D001), corrosivity (D002), chromium (D007), lead (D008), and mercury (D009). All waste generated in sampling and analysis efforts will be characterized and disposed of at an off-site disposal facility. It is estimated that up to three cubic meters of waste may be generated during sampling activities.

8.0 REFERENCES

C-AAC, 2015. QA-1, R.10:Actinide Analytical Chemistry Quality Assurance Program, Effective: 01/29/2015.

DOE 1995. *DOE Methods for Evaluating Environmental and Waste Management Samples*. DOE/EM-0089T, Rev. 2. Prepared for the U.S. Department of Energy by Pacific Northwest Laboratory, Richland, Washington.

EPA 1986 and all approved updates. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*. EPA-SW-846, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, U.S. Government Printing Office, Washington, D.C.

EPA 2002a. *RCRA Waste Sampling Draft Technical Guidance Planning, Implementation, and Assessment*. EPA530-D-02-002, Office of Solid Waste, U.S. Environmental Protection Agency, Washington, D.C. August 2002.

EPA 2002b. *Guidance on Choosing a Sampling Design for Environmental Data Collection for Use in Developing a Quality Assurance Project Plan*. EPA/240/R-02/005, December 2002. (http://www.epa.gov/quality/qs-docs/g5s-final.pdf)

LANL 2015a. *Transmittal of Los Alamos National Laboratory Information Regarding Legacy Nitrate Salt Waste Containers*. ENV-DO-15-0007, LA-UR-15-20084, February 13, 2015.

LANL 2015b. *LANL Waste Management*. P409, Rev. 5. Los Alamos National Laboratory, Los Alamos, NM. July 2015.

NMED 2010 and all approved updates. Los Alamos National Laboratory Hazardous Waste Facility Permit. New Mexico Environment Department, Santa Fe, NM. November 2010.

Table 1

Constituents of Concern within Unremediated Nitrate Salt Waste Containers

Category	Specific Constituents
Metals	Silver, Arsenic, Barium, Cadmium, Chromium, Mercury, Lead, Selenium
Anions	Nitrate (NO_3-), Nitrite (NO_2-) Chloride (Cl-), Fluoride (F-), Sulfate (SO_4-), Oxalate (C_2O_{4-})
Other Major Elements	Sodium, Potassium, Aluminum, Calcium, Iron, Magnesium, Silica

Table 2

Summary of Analytical Methods

Analyte	Test Methods/ Instrumentation
Major and minor elements (metals)	Inductively coupled plasma atomic emissions spectroscopy (ICP-AES) and mass spectrometry (ICP-MS)
Anions	Ion chromatography
Radionuclides	Nondestructive assay by gamma spectroscopy, FRAM (a software analysis tool), and Spectral Nondestructive Assay Platform (SNAP)
pH	Potentiometric pH method
Moisture content	Loss on ignition 110 degrees Celsius (^{o}C)
Combustible content	Loss on ignition 600 ^{o}C
Major chemical composition and form	X-ray diffraction , micro X-ray fluorescence and thermogravimetry, differential scanning calorimetry mass spectrometry (TGA-DSC-MS) and other methods as determined useful depending on the sample

Table 3

Sample Selection for Unremediated Nitrate Salt Waste Containers

Sample?	Drum ID	Container ID	Justification for/against Sampling	Waste Stream	RTR Date	RTR Operator Comments	Rigid / Lead Liners	Additional Comments	Location of Proposed Solid Samples	Location of Proposed Liquid Samples	Date Sealed	No. of Bags
Yes	5076	S793724	Selection based on generation date (earliest available date is 1979), identified discrepancy in the availability of liquids within the container, and only container with generator documentation that includes mention of "NaOH."	LA-CIN01.001-Cans	5/8/2014	85-gal. container; internal vented lead-lined 55-gal. container w/ vented 90-mL liner/lid; currently assigned to CIN01, matches, is S3000 solids in bags; prohibited liquid >1% of 85 gal., >3% of 55 gal., 1–2 gal. within waste (potentially partially absorbed)	Rigid / Lead	Lead liner pulled away a bit. Container is about 1/2 to 2/3 full. Before current RTR, liquids were identified as greater quantity. Paperwork states packaged with "filterable NaOH pellets."	Top and lower level of waste	Liquids observed at bottom of container	11/6/1979	11
	6544	S801676		LA-CIN01.001-Cans	5/8/2014	85-gal. container; internal unvented lead-lined 55-gal. container w/ unvented 90-mL liner/lid (>4 L sealed containers); currently assigned to CIN01, matches, is S3000 solids in bags; prohibited liquid >1% of 85 gal., >3% of 55 gal., 1–2 gal. within waste (potentially partially absorbed)	Rigid / Lead	2/3 full. Lead liner in decent shape (a little bent over). Liquids are a bottom of the container.			4/15/1980	14
	5274	70069 (S802701)	The top of this container was sampled in April 2014.	LA-MIN02-V.001	5/7/2014	85-gal. container; 1 L of liquid at 20 in. in bag S3000 lead-lined	Rigid / Lead	Passed with acceptable amount of liquid. About 1/2 full and liquid appears to be on top.			2/29/1980	9
Yes	6513	S802739	Chosen as one of two containers representing generation date 1980, and the location of the liquid within the container appears to be at the top of the waste and may be more accessible for sampling.	LA-CIN01.001-Cans	5/8/2014	85-gal. container; internal vented lead-lined 55-gal. container w/ vented 90-mL liner/lid; currently assigned to CIN01, matches, is S3000 solids in bags; prohibited liquid >1% of 85 gal., >3% of 55 gal., 1–2 gal. within waste (potentially partially absorbed)	Rigid / Lead	1/2 full. Lead liner is in decent condition. There looks to be liquid at the top of the container and partially absorbed at the bottom of the container.	Top and lower level of waste	Liquid appears to be located at the top of the waste and may be easier to access. There is also partially absorbed liquid at the bottom of the container.	3/19/1980	10
	6201	S802833		LA-CIN01.001-Cans	5/9/2014	85-gal./55-gal. containers; poly liner (vented) lead-lined pockets of liquid approx. 5 gal. of liquids throughout	Rigid / Lead	Liquid is partially absorbed and there are quite a few contents within the container.			5/9/1980	12
	6725	S803078		LA-CIN01.001-Cans	5/8/2014	85-gal. container; internal vented lead-lined 55-gal. container w/ vented 90-mL liner/lid; currently assigned to CIN01, matches, is S3000 solids in bags; prohibited liquid >1% of 85 gal., >3% of 55 gal., 2–3 gal. in bag folds from middle to bottom of the container	Rigid / Lead	2/3 full. Lead liner is crumpled but not bad. Liquids from the middle to the bottom of the container.			7/25/1980	18

Table 3

Sample Selection for Unremediated Nitrate Salt Waste Containers

Sample?	Drum ID	Container ID	Justification for/against Sampling	Waste Stream	RTR Date	RTR Operator Comments	Rigid / Lead Liners	Additional Comments	Location of Proposed Solid Samples	Location of Proposed Liquid Samples	Date Sealed	No. of Bags
	8031	S804948		LA-CIN01.001-Cans	5/8/2014	85-gal. container; internal vented lead-lined 55.-gal container w/ vented 90-mL liner/lid; currently assigned to CIN01, matches, is S3000 solids in bags; prohibited liquid >1% of 85 gal., >3% of 55 gal., 1–2 gal. within waste (potentially partially absorbed)	Rigid / Lead	1/2 full. Lead liner is falling a bit. Liquids are at the bottom of the container.			9/8/1980	4
	9577	S804995		LA-CIN01.001-Cans	5/7/2014	85-gal. container; S3000 no cans lead-lined, 2 gal. of liquid at bottom of container	Rigid / Lead	1/2 full. Liner bent. Liquid starts at 28 in. and goes all the way to the bottom.			11/18/1980	11
Yes	6789	S805051	Chosen as one of two containers representing generation date 1980, and the location of the liquids within the container appear to be accessible for sampling.	LA-CIN01.001-Cans	5/7/2014	85-gal. container; S3000 no cans lead-lined, 2 gal. of liquid from 24 in. to bottom of container	Rigid / Lead	Liner in good condition. 2/3 full. Liquid in top bag and at bottom of container.	Top and lower level of waste	Liquid appears to be located at the top of the waste and may be easier to access.	9/3/1980	13
	9263	S805289		LA-CIN01.001-Cans	5/7/2014	85-gal. container; S3000 no cans lead-lined, 3 gal. of liquid at bottom of container	Rigid / Lead	2/3 full. Liquid at 21 in. and more at the bottom of the container.			11/25/1980	7
	9884	S813385		LA-CIN01.001-Cans	5/7/2014	85-gal container; S3000, no cans, lead-lined 1 gal. of liquid at 24 in.	Rigid / Lead	1/2 full. Liquid from 24 in. to all the way down the container.			4/28/1981	7
Yes	9886	70072 (S813389)	Originally chosen as an alternate. Please see comment to S853714 below. Potential for sampling identified because of deterioration of lead liner and abundance of liquid. S813545 determined to be a better candidate because the liquid looks like it is easier to access in S813545 because the top of this container was sampled in 2014 and because S813545 contains more bags of salts.	LA-CIN01.001-Cans	5/7/2014	85-gal. container; S3000, no cans lead-lined 5 gal. of liquid at bottom of container	Rigid / Lead	2/3 full (starts at 16 in.). Crumpled liner.	Top, mid-level, and lower level of waste	Liquid can be found at ~24 in. on ruler and more liquids are located at the bottom of container.	4/29/1981	4
Yes	8808	S813545	Chosen for representing generation date 1981 and while it looks less full than container S813389, characterization documentation states that it contains more bags of salts. Also represents a container with a crumpled liner and contains quite a bit of liquid that may be accessible for sampling.	LA-CIN01.001-Cans	5/8/2014	85-gal. container; internal unvented lead-lined 55-gal. container w/ unvented 90-mL liner/lid (>4-L sealed containers); currently assigned to CIN01, matches, is S3000 solids in bags; prohibited liquid >1% of 85 gal., >3% of 55 gal., 3–4 gal. within liner	Rigid / Lead	1/3 full. Liner is crumpled on top of waste. Liquid appears up the side of the waste.	Top and lower level of waste	Liquid appears to be located all the way up the side of the waste.	1/22/1981	7

Table 3

Sample Selection for Unremediated Nitrate Salt Waste Containers

Sample?	Drum ID	Container ID	Justification for/against Sampling	Waste Stream	RTR Date	RTR Operator Comments	Rigid / Lead Liners	Additional Comments	Location of Proposed Solid Samples	Location of Proposed Liquid Samples	Date Sealed	No. of Bags
	10094	S816434		LA-CIN01.001-Cans	5/7/2014	85; S3000 no cans lead-lined, 1 gal. of liquid at bottom of container	Rigid / Lead	2/3 full. Liquid in the middle of the container in pockets as well as at the bottom of the container.			7/27/1981	4
Yes	10934	S816810	Chosen for representing generation date 1981, location of liquid within the container, and radiological representation.	LA-CIN01.001-Cans	5/9/2014	85-gal./55-gal. containers; poly liner (vented) lead-lined pockets of liquid approx. 5 gal. at 24 in.	Rigid / Lead	2/3 full. Lead liner in good shape. Pockets of liquid from 24 in. all the way down to bottom of container.	Top, mid-level, and lower-level of waste	Appears to contain many pockets of liquid within the last 10–12 in. of the container.	9/22/1981	3
	11377	S818435		LA-CIN01.001-Cans	5/7/2014	85-gal. containers; S3000 no cans lead-lined 5 gal. of liquid at bottom of container. Batch closed.	Rigid / Lead	Liquid between rigid liner and 55-gal. container. 1/2 to 2/3 full. Liner in decent shape.			12/16/1981	6
	11323	S822599		LA-CIN01.001-Cans	5/8/2014	85-gal. container; internal vented lead-lined 55-gal. container w/ vented 90-mL liner/lid; currently assigned to CIN01, matches, is S3000 solids in bags; prohibited liquid >1% of 85 gal., >3% of 55 gal., 3–4 gal. in bag folds from middle to bottom of the container and within liner	Rigid / Lead	2/3 full. Lead liner in good shape. Does not appear to be much at the bottom of the container.			1/8/1982	6
Yes	11374	S822713	Chosen for representing generation date 1982, liquid amount and location, number of bags of salt, crumpled lead liner, and radiological representation.	LA-CIN01.001-Cans	5/8/2014	85-gal. container; internal vented lead-lined 55-gal. container w/ vented 90-mL liner/lid; currently assigned to CIN01, matches, is S3000 solids in bags; Prohibited liquid >1% of 85 gal., >3% of 55 gal., 4–5 gal. within liner	Rigid / Lead	~2/3 full. Operator commented on lead being deteriorated. Lead liner is crumpled a bit. Liquid is almost all the way up the side of the container.	Top, mid-level, and lower level of waste	Liquid can be observed almost all the way up the side of the container.	1/22/1982	6
Alternate	11326	S822844	Potential for sampling identified because of liquid content; however, operator reports more liquid than can be observed in RTR. S825879 determined to be a better candidate because liquids appear more accessible.	LA-CIN01.001-Cans	5/9/2014	85-gal./55-gal. containers; poly liner (vented) lead-lined pockets of liquid approx. 15 gal. at 20 in.	Rigid / Lead	Operator commented some of the liquid appears to be a sludge substance. 2/3 full. Lead liner in decent shape. Looks like pockets of liquid at 24 in. and 28 in. Seems like the operator identified a lot more liquid that can be seen in the video.			2/22/1982	3
	9736	S823124		LA-CIN01.001-Cans	5/8/2014	85-gal. container; internal vented lead-lined 55-gal. container; currently assigned to CIN01, matches, is S3000 solids in cans	Lead Only	No bags only cans.			4/15/1982	2
	9920	S823184		LA-CIN01.001-Cans	5/9/2014	85-gal./55-gal. containers; poly liner (vented) lead-lined pockets of liquid approx. 5 gal. at 24 in.	Rigid / Lead	1/2 full. Appears dry.			4/28/1982	5

Table 3

Sample Selection for Unremediated Nitrate Salt Waste Containers

Sample?	Drum ID	Container ID	Justification for/against Sampling	Waste Stream	RTR Date	RTR Operator Comments	Rigid / Lead Liners	Additional Comments	Location of Proposed Solid Samples	Location of Proposed Liquid Samples	Date Sealed	No. of Bags
	13748	S825878		LA-CIN01.001-Cans	5/8/2014	85-gal. container; internal vented lead--lined 55-gal. container w/ vented 90-mL liner/lid; currently assigned to CIN01, matches, is S3000 solids in bags	Rigid / Lead	1/2 full. Liner in good shape. Appears dry.			12/15/1982	5
Yes	12074	S825879	Chosen as second representing generation date 1982 because it contains liquids, unlike S823124, S823184, and S825878. Additionally, the liquids may be accessible and not sludge-like (S822844).	LA-CIN01.001-Cans	5/7/2014	85-gal. container; S3000 no cans lead-lined 2 L of liquid at bottom of container	Rigid / Lead	2/3 full. Small amount of liquid 24 in. to the bottom of the container.	Top and lower level of waste	Liquid may be accessible from the side of the container. Appears to be within last 10–12 in. or so of the container.	12/15/1982	4
	15265	S842446		LA-CIN01.001-Cans	5/8/2014	85-gal. container; internal vented 55-gal. container w/ no liner; Currently assigned to CIN01, matches, is S3000 solids in cans	No Liners	No bags only cans.			2/3/1984	
Potential	15303	69907 (S844602)	Container will be evaluated by RTR to determine if sample(s) can safely be collected from this container.	LA-MIN02-V.001	2/21/2013	85-gal. container; >1% and >3% prohibited liquid, approx. 4.5 gal.		Needs High energy RTR (HERTR) Prescreen & Video. No video available.			5/11/1984	
Potential	19661	69904 (S851415)	Container will be evaluated by RTR to determine if sample(s) can safely be collected from this container.	LA-MIN02-V.001	7/12/2012	Not in acceptable knowledge. Impenetrable (lead liner). Internal vented 55-gal./90-mL rigid liner. >1% liquid in 90-mL liner.	Rigid / Lead	Needs HERTR Prescreen & Video. No video available.			1/2/1985	10
	20603	S853714	Originally chosen as representing generation date 1985 because container S851415 has more unknown variables associated with sampling. Removed from selection because the container is unvented and venting operations at the facility cannot be conducted at this time.	LA-CIN01.001-Cans	5/8/2014	85-gal. container; internal unvented lead-lined 55 gal. container w/ unvented 90-mL liner/lid (>4 L sealed containers); currently assigned to CIN01, matches, is S3000 solids in bags; prohibited liquid >1% of 85 gal., >3% of 55 gal., 3–4 gal. within liner	Rigid / Lead	Operator comments on liquid appearing to be potentially partially absorbed. 1/2 full. Liner in good condition. Partially absorbed liquids in folds.			6/21/1985	12
	21049	S862888		LA-CIN01.001-Cans	5/8/2014	85-gal. container; internal vented lead-lined 55-gal. container w/ vented 90-mL liner/lid; currently assigned to CIN01, matches, is S3000 solids in bags; prohibited liquid >1% of 85 gal., >3% of 55 gal., 2–3 gal. in bag folds in bottom third of the container	Rigid / Lead	2/3 full. Liner looks good. Liquids appear to be at the bottom of the container.			5/8/1986	11

Table 3

Sample Selection for Unremediated Nitrate Salt Waste Containers

Sample?	Drum ID	Container ID	Justification for/against Sampling	Waste Stream	RTR Date	RTR Operator Comments	Rigid / Lead Liners	Additional Comments	Location of Proposed Solid Samples	Location of Proposed Liquid Samples	Date Sealed	No. of Bags
Yes	21268	S864213	Chosen as representation with a generation date of 1986 because there appears to be more liquid within this container than S862888.	LA-CIN01.001-Cans	5/1/2014	85-gal. container; internal vented lead-lined 55-gal. container w/ unvented 90-mL liner; prohibited item, >4 L sealed container; 90-mL unvented liner; prohibited liquid 5+ gal. within liner	Rigid / Lead	Lead appears to be deteriorated. 2/3 full. Lots of liquid up to about half way point.	Top, mid-level, and bottom-level	Liquids located from approximately half-way down the container to the bottom.	8/25/1986	11